

## Effects of Extent of Chlorination, Extraction Rate, and Particle Size Reduction on Flour and Gluten Functionality Explored by Solvent Retention Capacity (SRC) and Mixograph

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Flour chlorination has been used for producing cake flour since the 1930s (Gough et al 1978). In addition to chlorination, low extraction and postmilling to reduce particle size are preferred treatments for producing cake flour. Chlorination affects all flour components, including starch, proteins, lipids, and pentosans. It results in enhanced starch pasting, increased hydrophobicity of starch granule surface protein, decreased gluten protein strength, and increased water-holding capacity (Holme 1962; Cole 1970; Kissel 1971; Kulp et al 1972; Kissel et al 1979; Gaines and Donelson 1982; Huang et al 1982a,b; Donelson et al 1984; Telloke 1985; Seguchi 1987; Conforti et al 1993; Duviau et al 1996; Baldwin et al 1997).

For analyzing the quality and functionality of soft wheat flours, the solvent retention capacity (SRC) method was conceived and developed by Slade and Levine (1994) and implemented as an AACC Approved Method (Gaines 2000). The SRC test is a solvation assay for flours based on the swelling behavior of polymer networks in selective diagnostic solvents using water, 5% lactic acid, 5% sodium carbonate, and 50% sucrose. Each solvent can diagnose the functional contribution of each flour component: water SRC is related to overall water-holding capacity by all network-forming components; lactic acid SRC is related to glutenin network forming and gluten strength; sodium carbonate SRC is related to damaged starch contribution; and sucrose SRC is related to water-accessible pentosans (arabinoxylans). The SRC method is much simpler and less labor-intensive than cake baking, which is traditionally used for testing cake flour quality. Numerous researchers have successfully applied SRC testing to soft wheat breeding programs and milling and baking quality evaluations of soft wheat and triticale flours (Guttieri et al 2001, 2002; Bettge et al 2002; Guttieri and Souza 2003; Ram and Singh 2003; Roccia et al 2006; Kweon et al 2009). Based on the demonstrated utility of SRC testing as a valuable tool for evaluating soft wheat quality, Xiao et al (2006) recently applied SRC testing for evaluation of hard winter wheat quality for breadmaking and showed that the lactic acid SRC value correlated with the quality of gluten protein relating to baked loaf volume over a wide range of flour protein contents. Also, lactic acid SRC results were significantly correlated to SDS-sedimentation volume; the test was reliable in predicting the loaf volume of breads from HWW flours with similar protein contents.

One of the major uses of soft wheat flour is for cake production, but SRC analysis of chlorinated cake flours has not been reported previously. High protein flour is not as preferred for cake production. Chlorination weakens the gluten strength of flour and

is accompanied by substantial changes in the amino acids of gluten (Ewart 1968). Ram et al (2005) reported that lactic acid SRC exhibited significant positive correlations with farinograph and mixograph parameters related to gluten strength. In the present study, the effects of extraction rate, extent of chlorination, and particle size reduction on flour and gluten functionality were explored using SRC and mixograph. We introduced a new predictive SRC parameter: the ratio of lactic acid (LA)/[sodium carbonate (NaC) + sucrose (Suc)] SRC values, to describe the overall performance of glutenin in the environment of other modulating networks (L. Slade, *personal communication*).

### MATERIALS AND METHODS

#### Wheat and Milling

Croplan 594W (soft red winter wheat grown in Ohio in 2004) was cleaned, tempered to 14% moisture, and milled with a Miag Multomat mill, to prepare straight-grade flour (SG) and low-extraction flour (Low X, 55% milling yield) samples. Parts of the low-extraction flour sample were further processed on an Alpine Kolloplex model 160-Z pin mill at 10,250 rpm to reduce particle size. The pin-milled flour was referred to as postmilled flour. For chlorination of SG, Low X, and postmilled flours,  $\approx 1$  lb of each flour was treated with chlorine gas to reach specific target pH levels (5.2, 4.9, 4.6, 4.3, 4.0, and 3.7) (Mennel Milling, Roanoke, VA). Moisture, ash, and protein contents were measured using AACC Approved Methods 44-16, 08-01, and 46-30, respectively. The pH levels for all flour samples were measured on 10% flour slurries using a pH meter (Fisher Scientific, Pittsburgh, PA).

#### Particle Size Analysis

Particle size distribution for each flour sample (50 g) was determined by Ro-tap sieve shaker with 105, 75, and 45  $\mu$ m meshes; the run time for the shaker was 40 min. Each sieve was used over a blank sieve with 0.5 in. wire cloth and with high bounce “screw balls” to avoid flour agglomeration due to electrostatics. The weight percent of each flour particle size fraction was calculated based on the fraction weight retained over each sieve.

#### Solvent Retention Capacity (SRC)

All flour samples were analyzed by SRC testing. SRC testing with four solvents followed Approved Method 56-11 (AACC International 2000). Flour samples (5 g) were suspended in 25 g of deionized water, 5% (w/w) lactic acid, 5% (w/w) sodium carbonate, and 50% (w/w) sucrose, and hydrated for 20 min with shaking at 5-min intervals. The hydrated flour slurries were centrifuged at  $1,000 \times g$  for 15 min, and the supernatants were drained. Each pellet was weighed and the SRC (%) for each sample was calculated according to AACC Approved Method 56-11.

#### Mixograph

Mixograph analysis of flour was done to evaluate the gluten strength of chlorinated flour samples. Representative SG and Low

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X flour samples were selected and tested on a mixograph (TMCO National) using 10 g of sample according to Approved Method 54-40A (AACC International 2000). The added water level was determined based on water SRC values; the total mixing time was 7 min for all samples.

## RESULTS AND DISCUSSION

### Proximate Analyses

The soft red wheat cultivar, Croplan 594W, exhibited excellent milling performance. Its low kernel hardness enabled a 33% break flour yield and the ease of separation of endosperm from bran resulted in a 74% milling yield (based on cleaned wheat). Moisture contents of the unchlorinated flour samples were  $\approx 13.3$ –13.6%, except for the postmilled flour sample (12.4% moisture content). For the latter, the results indicated that moisture loss occurred during the pin-milling process. The SG flour contained 7.6% protein and 0.40% ash (vs. 1.677% wheat ash), whereas both the Low X and postmilled flours contained 6.9% protein and 0.32% ash.

The pH levels of the Low X and postmilled flours (both pH 5.7) were lower than that of the SG flour (pH 5.9) due to decreased bran contamination. Li and Posner (1989) reported the relationship ( $r = 0.987$ ) between ash content and flour extraction level, whereby the ash content in flour increased as the level of extraction was raised. Posner and Hibbs (1997) explained the increase in ash content caused by the incorporation of endosperm close to the bran and the aleurone layer, which inherently contain higher levels of minerals.

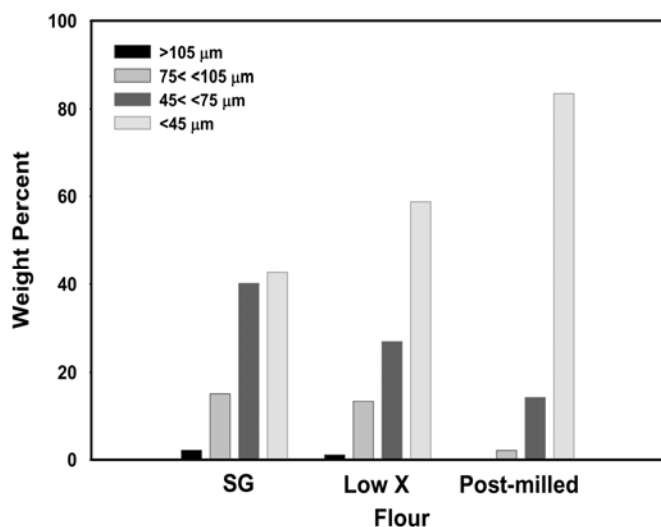


Fig. 1. Weight percents for particle size distribution of Croplan 594W flour samples measured using a Ro-tap sieve shaker.

### Particle Size Distribution

Weight percents for flour particle size distributions measured with the Ro-tap sieve shaker are shown in Fig. 1. The SG flour contained larger particles than the Low X flour, which showed bigger portions in the  $>45$ - $\mu\text{m}$  fractions, and a smaller portion in the  $<45$ - $\mu\text{m}$  fraction. Compared with the SG and Low X flours, the postmilled flour had significantly smaller particles, with a dramatic decrease in the  $>45$ - $\mu\text{m}$  fractions and a dramatic increase in the  $<45$ - $\mu\text{m}$  fraction. Chaudhary et al (1981) showed that particle size decreased by pin-milling, and the effect of pin-milling differed with wheat class. Gaines (1985) also reported that the mean volume diameters of 83 soft red and white wheat flour samples were approximately halved after pin-milling.

### SRC

SRC results for the flour samples are shown in Fig. 2. The pattern of SRC values for the unchlorinated SG flour in four solvents (47.8% water, 82.9% lactic acid, 64.5% sodium carbonate, and 83.4% sucrose) represents an excellent quality of soft wheat flour because the flour contained low levels of damaged starch and arabinoxylans, and showed absence of excessive glutenin functionality. Compared with the SG flour, the SRC values for the Low X flour showed a decrease in sucrose SRC value due to less arabinoxylans resulting from less bran contamination, a decrease in sodium carbonate SRC value due to less damaged starch, and an increase in lactic acid SRC value due to greater gluten strength resulting from a larger endosperm proportion. The postmilled flour showed an increased sodium carbonate SRC value due to increased damaged starch resulting from decreased particle size by pin-milling compared with the Low X flour, but the sodium carbonate SRC value for the postmilled flour was still lower than that for the SG flour. Our results supported well the report by Gaines and Donelson (1985) that postmilling cake flour treatments reduced particle size and increased starch damage.

For the chlorinated SG, Low X, and postmilled flours, lactic acid SRC values decreased but water, sodium carbonate, and sucrose SRC values increased with increasing extent of chlorination. The extent of decrease or increase was different in each solvent. The significant decrease in lactic acid SRC values reflected the decrease in gluten strength caused by chlorination. Sodium carbonate SRC values showed a relatively slight increase, which suggested a minimal effect on the swelling properties of starch granules due to chlorination. In particular, sucrose SRC values increased dramatically at less than pH 4.5, reflecting a bigger change in arabinoxylans for heavily chlorinated flour. These results suggested that oxidative gelation of arabinoxylans by an oxidizing agent such as chlorine gas could have occurred; there is a need for further investigation of this possibility. Overall, the SRC results revealed dramatic effects of milling extraction rate and chlorination extent, but less significant effects of additional milling to reduce flour particle size. The chlorination of cake flour is more effective in reducing the lactic acid SRC value

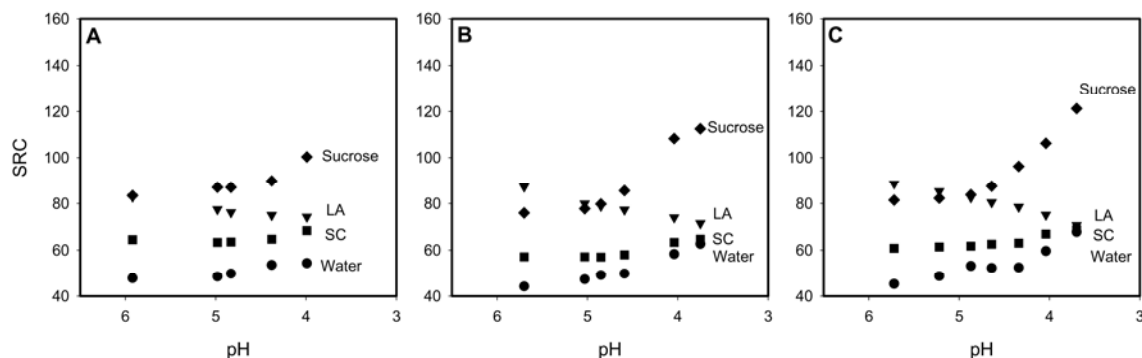
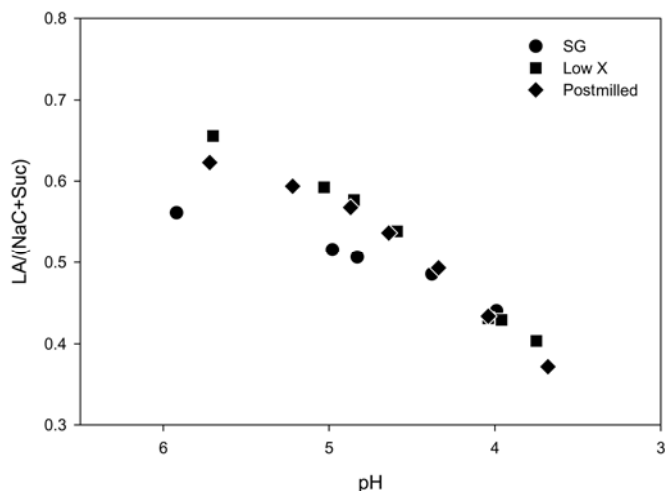


Fig. 2. SRC profiles for the SG (A), Low X (B), and postmilled (C) flours from Croplan 594W.

in flour with finer particle size than coarser particle size. The lactic acid SRC value declined by 7.5% for each decline in one pH unit in the pin-milled Low X flour. This was in contrast to a 7.0% decline for the Low X flour and a 6.7% decline for the SG flour. The slopes across the pH gradient for sucrose SRC and sodium carbonate SRC were not significantly different among the flours, but there was a small difference in the slopes for water SRC.

Although the SRC values for the different solvents can give information on the individual contribution of each functional component, all the values are, to some extent, convoluted with each other due to the four solvents all being water-basis solvents. To deconvolute the contributions of gluten strength from damaged starch and arabinoxylans, and to describe the overall performance of glutenin in the environment of other modulating networks, we introduced a new predictive SRC parameter: the ratio of LA/(NaC + Suc) SRC values (L. Slade, *personal communication*) as plotted in Fig. 3. The SG flour showed a lower ratio than the Low X and postmilled flours, which suggested weaker gluten strength. The ratio of LA/(NaC + Suc) SRC values decreased significantly with increasing extent of chlorination and the effect was greater when the milling yield decreased from 74 to 55%. Kweon et al (2009) reported the benefit of this SRC ratio in a tempering study; that is, the ratio can be used to determine optimum flour extraction to elevate gluten strength while minimizing overall water absorption.



**Fig. 3.** Ratio of LA/(NaC + Suc) SRC values for Croplan 594W flour samples with different extents of chlorination.

## Mixograph

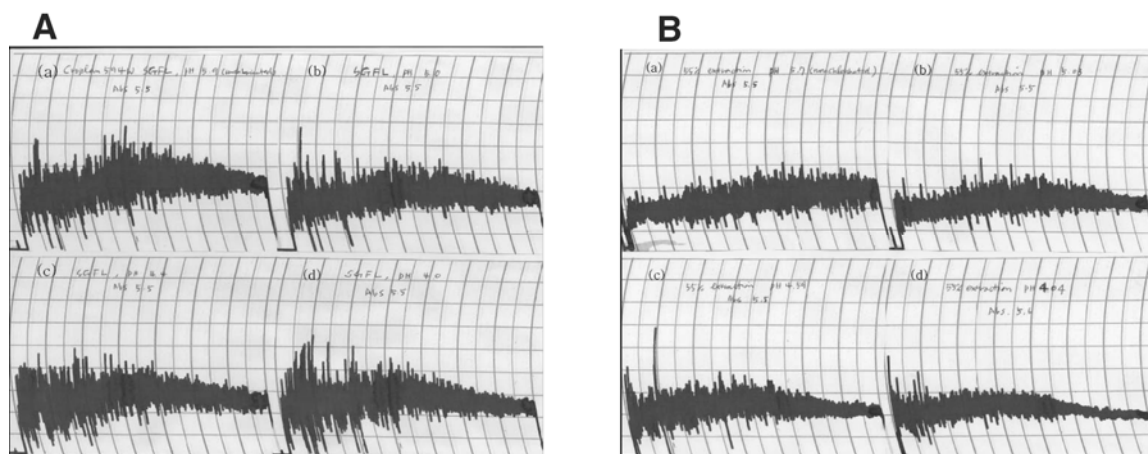
Mixograph results for the SG and Low X flours are presented in Fig. 4. For the unchlorinated flours, bandwidth for the Low X after peak (after  $\approx 4$  min) in Fig. 4B (a) was larger than that for the SG flour in Fig. 4A (a) due to increased gluten strength from a larger portion of endosperm and a greater gluten protein content. For the chlorinated flours with increasing extent of chlorination, the mixograph bandwidth after peak became narrower, indicating reduced gluten strength. The effect was much more evident for the Low X flour (Fig. 4B), compared with the SG flour (Fig. 4A). These mixograph results matched well with the corresponding decrease in lactic acid SRC values, as well as in the ratio LA/(NaC + Suc). However, the ratio LA/(NaC + Suc) SRC values appeared to be a better predictor of gluten functionality to explain the smaller change for the SG flour than for the Low X flour, as demonstrated by mixography. Ram et al (2005) reported the relationship between SRC and mixograph for 192 wheat genotypes; lactic acid SRC showed a significant positive correlation with mixograph peak time ( $P < 0.001$ ) and peak dough resistance ( $P < 0.001$ ).

## CONCLUSIONS

Chlorination is an essential soft wheat flour treatment for production of high-ratio cakes in the United States, frequently coupled with a postmilling treatment to reduce flour particle size. The effects of extent of chlorination, extraction rate, and particle size reduction on flour and gluten functionality have been explored by SRC and mixograph for the soft wheat cultivar, Croplan 594W. The SRC results showed dramatic effects of milling extraction rate and chlorination extent, but less significant effects of additional milling to reduce particle size. With increasing extent of chlorination, SRC analysis showed increases in water, sodium carbonate and sucrose SRC values, but a decrease in lactic acid SRC values. Although lactic acid SRC is sufficient to monitor glutenin network formation, our new SRC parameter, the ratio of LA/(NaC + Suc) SRC values, is an even better predictor for gluten functionality in the environment of other modulating networks, as demonstrated by mixography. The ratio LA/(NaC + Suc) SRC values decreased significantly with increasing extent of chlorination and the effect was greater when the milling yield decreased from 74 to 55%.

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**Fig. 4.** Mixograph profiles for SG and Low X flours of Croplan 594W. **A**, SG flour, (a) pH 5.9, (b) pH 5.0, (c) pH 4.4, (d) pH 4.0; **B**, Low X flour, (a) pH 5.7, (b) pH 5.0, (c) pH 4.5, (d) pH 4.0.

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